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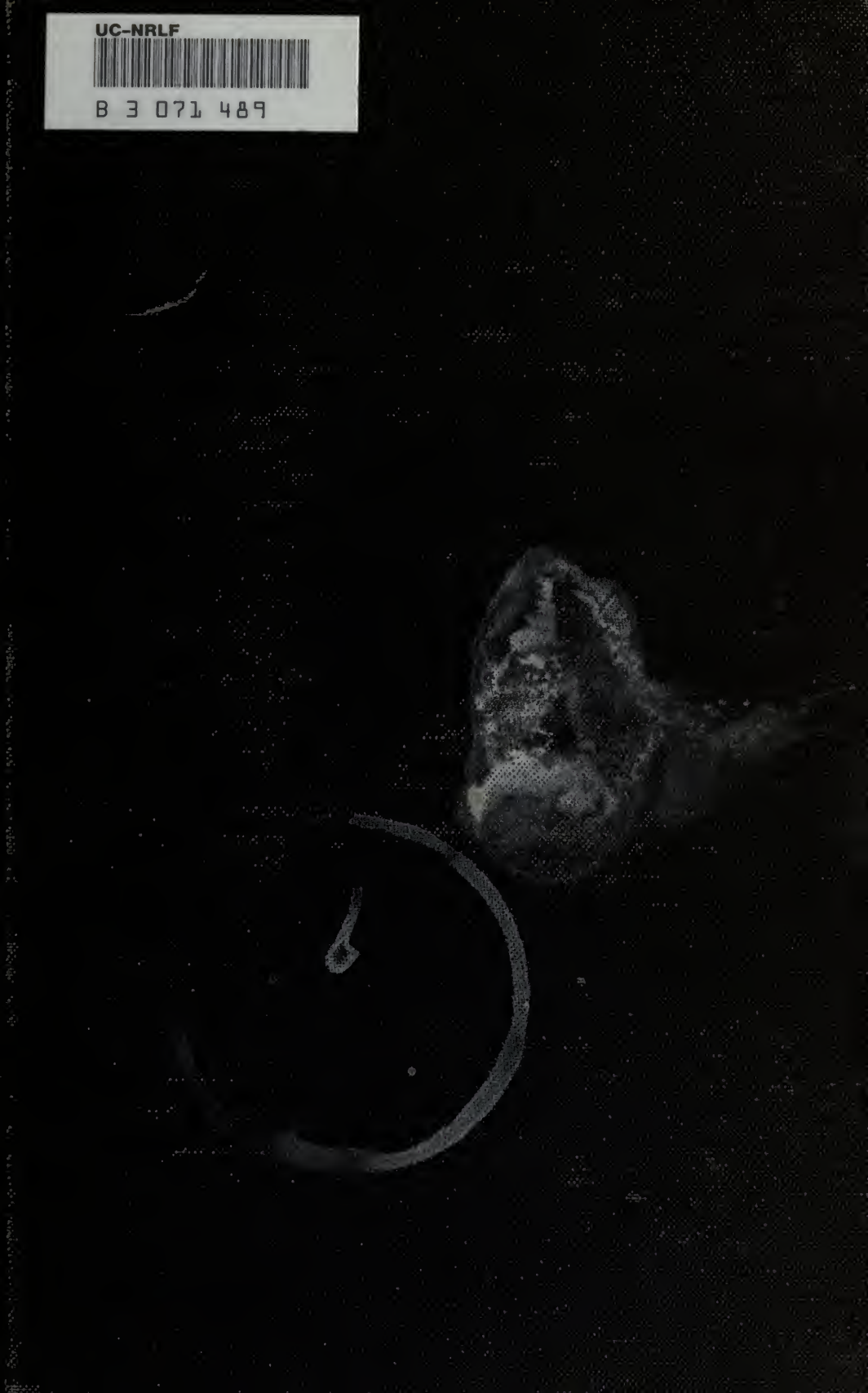

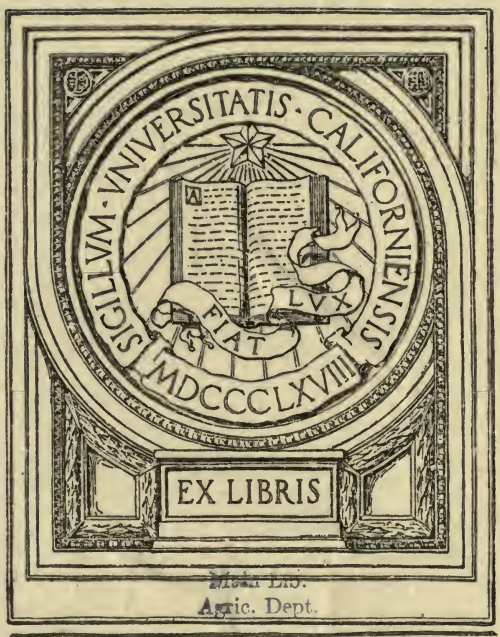
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BUREAU OF CHEMISTRY—Circular No. 80.

H. W. WILEY, Chief of Bureau.

TWO NEW PIECES OF CHEMICAL APPARATUS.

I. Apparatus for the continuous extraction of liquids with immiscible solvents lighter than water.
II. Apparatus for quantitative reactions which depend on the measurement of an evolved gas.

R. F. Bacon and P. B. Dunbar,

Assistant chemists, Division of Foods.

I. CONTINUOUS LIQUID EXTRACTOR.

This apparatus was designed originally for use in the extraction of lactic acid from ketchup and other fruit products. Its principal advantages are compactness, the elimination of ground joints and stoppers, and practically complete condensation. Seven of the extractors may easily be placed side by side on a 24-inch hot plate. They have been made for the authors at a cost of $1.50 each, but may be constructed in the laboratory by anyone having ordinary skill in glass blowing.

The apparatus consists of four parts: (1) A jacket flask; (2) an extractor thimble; (3) an ordinary Gooch funnel; (4) a condenser.

(1) The jacket flask (fig. 1, A) is made of glass tubing 2 inches in diameter and approximately one-sixteenth of an inch thick; it is 20.5 inches long and is enlarged to a diameter of about 3 inches at its lower or sealed end, as shown in the illustration. (2) The extraction thimble (fig. 1, B) is an ordinary test tube having a diameter of 1.5 inches and a capacity of 100 cc when filled to within 1.5 inches of the top. One-fourth of an inch from its top and on opposite sides of the tube are placed two holes about one-fourth inch in diameter. (3) The Gooch crucible funnel is figure 1, C; those used in this laboratory are 8.5 inches long. When dense liquids are to be extracted it is sometimes necessary to increase the length of the funnel. The lower end of the stem is ground at an angle of 45 degrees. (4) The condenser (fig. 1, D) is designed to hang loosely in the jacket flask. Its details are evident from the drawing. A simpler form of condenser, which is just as efficient, may be made by

sealing one end of a $1\frac{3}{4}$-inch tube and drawing it to a point. The open end is flared somewhat to permit the tube to hang in the jacket. The condenser tube is closed by a two-holed rubber stopper, through which pass the inflow and outflow tubes.

OPERATION.

Place from 100 to 150 cc of ether in the jacket flask (A); put the liquid to be extracted (100 cc) in the test tube (B), insert the funnel in the same tube and suspend it in the jacket flask, about 3 inches above the bottom, with a copper wire which passes through the holes in the test tube and is hooked over the rim of the jacket flask. Insert the condenser in the top of the jacket flask. The condensation is usually so perfect that no ether vapors escape into the room. The condensed ether drops from the point of the condenser into the funnel and is carried to the bottom of the test tube whence it flows up through the liquid and overflows at the top.

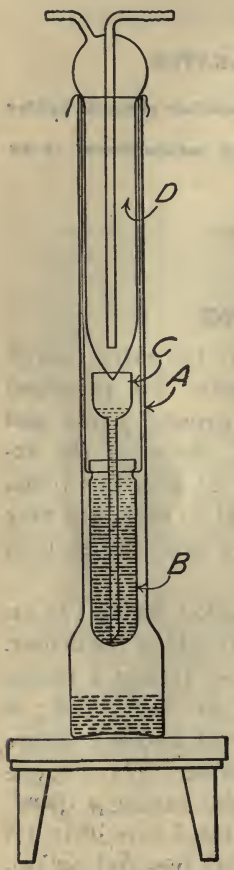

FIG. 1.—Continuous liquid extractor in operation.

The efficiency of the extractor may be increased by the use of a glass spiral attached to the stem of the Gooch funnel, as described by Kempf.[1]

II. APPARATUS FOR THE MEASUREMENT OF AN EVOLVED GAS.

The apparatus represented in figure 2 was devised to give in a compact and easily manipulated form an apparatus which will allow of the measurement of an evolved gas without first sweeping out all air or other indifferent gases. It consists of a graduated funnel tube (A), a reaction chamber (B), an absorption tube (C), filled with glass beads, a eudiometer (D), and a leveling tube (E). A heating coil may be wrapped around the reaction chamber (B) when desired. The absorption tube (C), which is sealed onto the eudiometer, fits into the reaction chamber (B) by means of a ground joint at (F).

The manner of using the apparatus may best be illustrated by one of the reactions which can be advantageously carried out in it. Spica[2] estimates citric acid from the carbon monoxid evolved by decomposing

[1] Chem. Ztg., 1910, 34: 1365; Chem. Abst., 1911, 5: 1350.
[2] Chem. Ztg., 1910, 34: 1141.

this acid with strong sulphuric acid at 100° C. He runs air-free carbon dioxid through a flask containing the citric acid until all air is displaced from the apparatus. He then adds concentrated sulphuric acid, heats to 100° C., drives over the evolved carbon monoxid with a stream of air-free carbon dioxid, and collects the gas in a eudiometer over a sodium hydrate solution. The method as carried out by him is accurate, but requires considerable care and attention to remove the air from the apparatus completely, to insure that all evolved carbon monoxid is swept over into the eudiometer, and to prevent the strong sodium hydrate solution from sucking back into the reaction flask containing carbon dioxid and sulphuric acid. In the apparatus proposed by us no attempt is made to remove the air before reaction, the volume of evolved gas being simply measured by the increase in volume of the total gases after reaction. Thus the Spica method as carried out in this apparatus is as follows:

Place about 0.2 gram of citric acid in the reaction chamber (B), open the stopcock (X) and bring the liquid (in this case a strong sodium hydrate solution) to the zero mark (G) in the eudiometer tube by raising or lowering the leveling tube (E). The air in the apparatus is then at atmospheric pressure. Close (X). To (A) add about 15 cc of concentrated sulphuric acid. Lower the leveling tube so that the air in the apparatus is under reduced pressure. By carefully opening the stopcock (X) run in exactly 10 cc of the sulphuric acid. Close (X) and heat to about 100° C. by means of the heating coil until the reaction is complete. Let the apparatus stand until the absorption of other gases (sulphur dioxid, etc.) is complete and it has reached room temperature. Bring the gas in the apparatus to atmospheric pressure by means of the leveling tube (E). The gas reading in the eudiometer minus 10 cc (for the added sulphuric acid) equals the volume of the evolved gas at the existing temperature and pressure. The apparatus has been used in this laboratory with accurate results to estimate citric acid by the Spica method and for the estimation of amino acids by the Van Slyke method,[1] which depends on the evolution of nitrogen by the action of nitrous acid on the amino acids. The apparatus is readily cleaned by taking it apart at the ground joint (F).

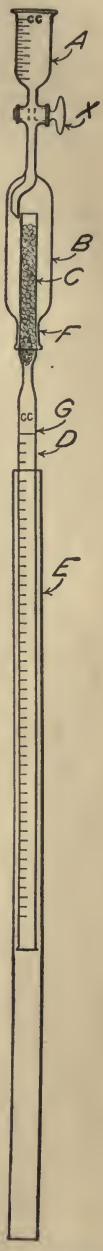

FIG. 2.—Apparatus for measurement of evolved gas.

[1] Ber. d. chem. Ges., 1910, 43(3) : 3170.

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